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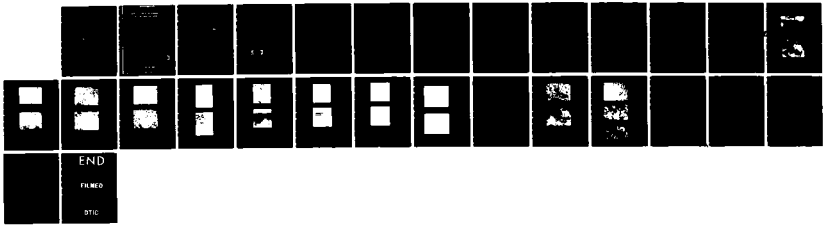

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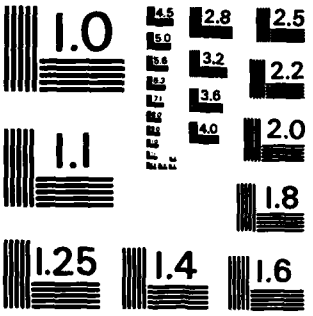

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APPLICATION OF RAPIDLY SOLIDIFIED ALLOYS

A. R. Cox
United Technologies Corporation
Pratt & Whitney Aircraft Group
Box 2691, West Palm Beach, Florida 33402

March 1979
Quarterly Report for Period 1 November 1978 to 1 February 1979

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Sponsored by
Defense Advanced Research Projects Agency

Prepared for
Air Force Materials Laboratories
Wright-Patterson AFB, Ohio 45433

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1 REPORT NUMBER	2. GOVT ACCESSION NO. AD-A148539	3 RECIPIENT'S CATALOG NUMBER
4 TITLE *(and Subtitle)* APPLICATION OF RAPIDLY SOLIDIFIED ALLOYS		5. TYPE OF REPORT & PERIOD COVERED Quarterly Report 1 November 1978 to 1 February 1979
		6. PERFORMING ORG. REPORT NUMBER FR-11690
7. AUTHOR(s) A. R. Cox, T. D. Tillman, J. W. Simon		8. CONTRACT OR GRANT NUMBER(s) F33615-76-C-5136
9. PERFORMING ORGANIZATION NAME AND ADDRESS United Technologies Corporation, Pratt & Whitney Aircraft Group, Government Products Division, Box 2691, West Palm Beach, Florida 33402		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Defense Advanced-Research Projects Agency, 1400 Wilson Boulevard, Arlington, Virginia 22209, (Dr. E. C. van Reuth)		12. REPORT DATE MARCH 1979
		13. NUMBER OF PAGES 25
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)* Air Force Materials Laboratories, Wright-Patterson Air Force Base, Ohio 45433, (Mr. A. Adair)		15. SECURITY CLASS. *(of this report)* Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for Public Release, Distribution Unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Powder Metallurgy, Rapid Solidification, Centrifugal Atomization, Convective Cooling, Aluminum Alloys, Iron Alloys

(Rapid Solidification Process)

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

This program is being conducted for the purpose of applying the principle of rapid solidification to aluminum and iron alloy powders and subsequent development of stronger alloy compositions for fan blade application (Al alloys) and higher speed bearing material (Fe alloys). Centrifugal atomization and forced convective cooling are being used to produce the fast-cooled powder. During this report period, adaptation of the RSR process to aluminum and iron systems was continued. Both Al and Fe alloys were produced and consolidated by direct extrusion. Hardness and mechanical testing were begun for initial evaluation of aluminum alloys.

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TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

SECTION I

INTRODUCTION

Rapid solidification of metal alloys has shown that distinct and dramatic changes in microstructure and crystal form can be attained beyond those possible by any known method of conventional solidification. These results are recognized by experts throughout the field of metallurgy as a means to achieve major improvements in metal strength, environmental compatibility, electrical properties, etc. Through the use of fast cooling, the following appears to be eminently possible: (1) stronger and more corrosion-resistant steels because of improved homogeneity, and (2) a new breed of aluminum, copper, and nickel alloys because of improved secondary phase dispersion.

An ARPA-sponsored program with the Pratt & Whitney Aircraft Group, Government Products Division (P&WA/Florida), has shown that by using the P&WA RSR process and equipment, it is possible to achieve rapid solidification in spherical powder under conditions which depict steady-state operations commensurate with production rates in excess of 1400 lb/hr. Further, this program has demonstrated that concurrent high product quality can be achieved and the resulting powder metal is in a form which can be readily handled and processed into useful shapes for subsequent application. No other method known to achieve similar rates of solidification can lay claim to these combined achievements.

The program has gone even further since it has demonstrated that controlled, rapid solidification can lead to a microcrystalline form, a condition which could possibly point the way to alloy homogeneity never before considered possible. It has also shown that a central rotary source can be used for liquid metal atomization into powder particles of sizes commensurate with average particle cooling rates of ~~10^5-10^{6} K/sec.~~ 100,000 – 1,000,000 K/sec.

This program is a modification to the Advanced Research Project Agency (ARPA) sponsored work which is directed toward superalloy development. Its purpose is to expand the scope of work in the field of rapid solidification from the exclusive study of superalloys to a study of aluminum, and iron base alloys. The specific objectives of this ~~added~~ effort are the development of an improved aluminum alloy suitable for V/STOL-A fan blades and an improved iron alloy suitable for rolling element bearings for advanced aircraft powerplants.

The program is a 36-month effort which begins with adaptation of the rapid solidification rate process to Al and Fe alloy systems and terminates with a payoff analysis of new materials as adapted to V/STOL-A and F100 advanced engine derivative requirements. This is the third technical report and covers the seventh through the ninth months of the program. It deals with adaptation of RSR processing to Al and Fe systems and the subsequent evaluation of these alloys.

Originator supplied Keywords include:

SECTION II

MATERIALS SELECTION

In the iron phase of activity, additional alloys were added to the first matrix, as shown in Table 1. The Fe alloys selected were primarily low in chromium, less than 9%, while molybdenum was varied from 2 to 8%, and carbon from 0.8 to 0.95%. Chromium was added to improve hardenability and, at the higher levels, corrosion resistance. Molybdenum was added to form the precipitate Mo_6C as a fine particle dispersion to improve hot hardness. The carbon level was varied to accommodate the changes in Mo and Cr concentration in order to increase the volume fraction of carbides for the purpose of improving wear resistance.

In the aluminum phase, no new additions were made to the original matrix. The primary matrix is also shown in Table 1. The matrix is being repeated with all inert handling to assess the effect on material properties. All powder made previously was air handled, but all subsequent powder will be handled inertly.

The first Al matrix was based on Co and Zn additions to a 7075 Al base. Cobalt additions were made for the purpose of forming an intermetallic compound, Co_2Al_9, which is insoluble up to the solidus temperature of the alloy. If present as a fine dispersion, the Co_2Al_9 is expected to control grain size and provide dispersion strengthening. High Zn contents can provide additional second phase for precipitation hardening.

TABLE 1. FIRST Al AND Fe MATRIX

Al Matrix*

	Cobalt			
Zn	0.8	1.6	2.4	3.2
5.6	X	X		X
7.0	X			X
8.4	X	X		X
9.8	X		X	

Fe Matrix*

		Molybdenum			
Cr	C	2	4	6	8
4	0.8	X	X (M-50)		X
9	0.8				
	9.5	X			X
14	0.8				
	1.1		X (EX-00007)	X	
19	0.8				
	1.25	X			X

1% V held constant

*Amounts are in wt %

SECTION III

CONVERSION AND CONSOLIDATION

Sixteen powder runs were made during this report period to gain more experience in atomization of the Al and Fe alloys under conditions of rapid solidification. These conversions were made with the compositions listed in Table 2. Data pertinent to the operations are tabulated in Table 3. Typically, yields of −140 mesh powder on the order of 50% or better were achieved. Difficulty was encountered with the iron alloys in that transfer tube cracking was evidenced in most of these runs and caused variable metal flowrates to adversely effect atomization. This problem is presently being solved by modification to the unit.

Powders from these runs were containerized for subsequent extrusions. Our procedure included initial hot outgassing at about 600°F (316°C) for the aluminum, 1000°F (538°C) for iron, at a vacuum level near 10^{-4} Torr. The aluminum alloys were containerized in 6061 cans, each nominally with a 3-in. (7.6 cm) dia by 7 in. (17.8 cm) long. The iron alloys were containerized in similar cans made from 304 stainless steel.

Twelve extrusions to produce test stock were done at AFML during this report period. Three of these extrusions were with Al compositions which are shown in Table 4. These were extruded at 15.5:1 reduction and 700°F (371°C), with specific information on each given in Table 5. The extrusions were visually sound with no evidence of cracking or other metalworking defects.

The remaining nine extrusions were with steel alloys. These alloys, likewise, had extrusion ratios of 15.5:1 and, in these cases, the extrusion temperature was varied from 1650°F (899°C) to 1750°F (954°C). Particulars on extrusion and composition are also given in Table 4 and 5. As with the Al, no difficulties were encountered.

TABLE 2. COMPOSITIONS OF ALLOYS CONVERTED TO POWDER

VM No.	Zn	MG	Cu	Co	Al	C	Cr	Mo	V	Si	Mn	Fe
678	5.6	2.5	1.0	0.8	Bal	—	—	—	—	—	—	—
649	—	—	—	—	—	0.81	4.0	4.25	1.0	0.15	0.25	Bal
650	—	—	—	—	—	1.15	14.75	4.0	1.2	0.30	0.45	Bal
678	—	—	—	—	—	0.80	4.0	2.0	1.0	0.15	0.20	Bal
680	—	—	—	—	—	0.95	9.0	2.0	1.0	0.15	0.20	Bal
681	—	—	—	—	—	0.80	4.10	4.25	1.0	0.15	0.20	Bal
701	—	—	—	—	—	0.80	4.0	8.0	1.0	0.15	0.20	Bal
702	—	—	—	—	—	0.95	9.0	8.0	1.0	0.15	0.20	Bal

TABLE 3. POWDER CONVERSIONS IN THIRD QUARTER

Run ID	VM No. ID	Nozzle Dia in. (cm)	Cup Speed Krpm	Cup Radius in. (cm)	Nozzle Temp °F (°C)	Pour Temp °F (°C)	T_{MP} °F (°C)	−140 Yield (%)	
52	650	0.125 (0.318)	24	5.250 (13.335)	2450 (1343)	2500 (1371)	2250 (1232)	55.3	
53	649	0.125 (0.318)	24	5.250 (13.335)	2450 (1343)	2670 (1466)	2420 (1327)	36.0	Cracked Nozzle
54	650	0.125 (0.318)	24	5.250 (13.335)	2430 (1332)	2550 (1399)	2330 (1277)	44.0	Cracked Nozzle
58	679	0.125 (0.318)	24	5.250 (13.335)	2540 (1393)	2700 (1482)	2450 (1343)	74.1	
59	680	0.125 (0.318	24	5.250 (13.335)	2525 (1385)	2700 (1482)	2390 (1310)	43.2	Cracked Nozzle
60	680	0.125 (0.318)	24	5.250 (13.335)	2475 (1357)	2700 (1482)	2380 (1304)	62.6	Cracked Nozzle
61	679	0.125 (0.318)	24	5.250 (13.335)	2530 (1388)	2700 (1482)	2400 (1316)	70.5	Slight Nozzle Crack
62	681	0.125 (0.318)	24	5.250 (13.335)	2560 (1404)	2730 (1499)	2400 (1316)	64.5	Cracked Nozzle
63	681	0.125 (0.318)	24	5.250 (13.335)	2540 (1393)	2700 (1482)	2400 (1316)	40.4	Slight Cracked Nozzle
70	701	0.125 (0.318)	(26) 24	5.250 (13.335)	2500 (1371)	2775 (1524)	2525 (1385)	55.1	Nozzle Split
71	701	0.125 (0.318)	24	5.250 (13.335)	2500 (1371)	2750 (1510)	2500 (1371)	41.9	Rubber Mounts and Turbine Balance
72	702	0.125 (0.318)	35	5.250 (13.335)	2500 (1371)	2675 (1468)	2425 (1329)	43.9	Nozzle Crack
73	702	0.125 (0.318)	35	5.250 (13.335)	2520 (1382)	2730 (1499)	2480 (1360)	75.9	
75	678	0.100 (0.254)	24	3.125 (7.938)	1460 (793)	1460 (793)	1160 (627)	46.7	
76	678	0.100 (0.254)	24	3.125 (7.938)	1500 (816)	1500 (816)	1160 (627)	46.3	
77	678	0.100 (0.254)	24	3.125 (7.938)	1350 (732)	1500 (816)	1175 (635)	62.7	

TABLE 4. COMPOSITIONS OF ALLOYS EXTRUDED

VM No.	Zn	Mg	Cu	Co	Al	C	Cr	Mo	V	Si	Mn	Fe
622	8.4	2.5	1.0	3.2	Bal							
630	9.8	2.5	1.0	0.8	Bal							
631	9.8	2.5	1.0	2.4	Bal							
649						0.81	4.0	4.25	1.0	0.15	0.25	Bal
650						1.15	14.75	4.0	1.2	0.30	0.45	Bal
679						0.80	4.0	2.0	1.0	0.15	0.20	Bal
680						0.95	9.0	2.0	1.0	0.15	0.20	Bal
681						0.80	4.10	4.25	1.0	0.15	0.20	Bal

TABLE 5. Al AND Fe EXTRUSION PARAMETERS

No.	*VM No.*	*Extrusion Temperature °F (°C)*	*Reduction Ratio*	*Break Through Tonnage*
40	622	700 (371)	15.5:1	280
44	630	700 (371)	15.5:1	260
45	631	700 (371)	15.5:1	230
52	650	1650 (899)	15.5:1	690
53	649	1750 (954)	15.5:1	670
54	650	1750 (954)	15.5:1	655
58	679	1750 (954)	15.5:1	620
59	680	1750 (954)	15.5:1	640
60	680	1650 (899)	15.5:1	690
61	679	1650 (899)	15.5:1	670
62	681	1650 (899)	15.5:1	670
63	681	1750 (954)	15.5:1	635

Note: All 60 deg conical dies

SECTION IV

MATERIALS EVALUATION

ALUMINUM ALLOYS

Metallographic examination of the Al extrusions was conducted for both the as-extruded and heat-treated conditions. The heat treatment was 870°F (466°C) for 1 hr with a water quench plus 250°F (121°C) age for 26 hr followed by an air cool. This is the T6 treatment for 7075 Al, chosen because of the similarities between 7075 and XSR alloys.

Figure 1 shows the microstructure of 7075-T6, which was procured as $^{3}/_{4}$ in. rod mill product for comparative purposes. The dark etching phase is reported to be $Cr_2 Mg_3 Al_{18}$. A second dark etching phase, $MgZn_2$, is also present but on a finer scale. The $MgZn_2$ is the primary aging phase for this alloy and should be for the rapidly quenched alloys as well. Figures 2 through 4 show the typical microstructures of the rapidly quenched alloys. Microprobe analyses of these alloys revealed little about the secondary phases present in these compositions. It did show that the dark gray phase had a higher Co and lower Cu concentration than the lighter phase. No positive identification of $MgZn_2$ was made with this technique, although it is known that the resolution of the microprobe prevents analysis of extremely fine phases. The Co_2Al_9, which was expected, was tentatively identified as one of the precipitates.

An attempt was made to use differential thermal analysis (DTA) for additional phase determination of the powders themselves. To us, this proved unsuccessful due to the complicated nature of these alloys. The actual traces obtained from each of the alloys shown in Figures 14 through 16 are included as Section VI of this report for possible reader interest. STEM techniques are being planned in the continuing effort of phase identification.

Metallographic evaluation revealed significant segregation of the Co in both the as-extruded and heat-treated condition, as shown in Figure 5. Concurrent analysis of the aluminum powder was made to determine if the segregation was due to extrusion or due to the atomization cycle itself. Although there have been no problems in the production of the powder, there is evidence from this study that the desired second phase dispersion of Co_2Al_9 was not being achieved because of melt conditions associated with the atomization cycle. Figure 6 is an SEM micrograph and the corresponding X-ray scan which shows a high concentration of Co in individual powder particles as compared to other alloying elements, such as Zn, which is ten times as abundant as Co in this alloy. Close examination of the powder particle (Figure 7) revealed that the second phase is approximately the Co_2Al_9 composition and that the small amount of iron which was observed, is present primarily as an impurity. Figure 8 is a content mapping for Co and the corresponding SEM micrograph. The white spots indicate the presence of Co and reveal its segregation to a single particle within the field of view. The SEM micrographs shown are from a 0.8% Co alloy and the segregation to single particles is a strong indication of Co segregation prior to the atomization. A comparison of the 0.8% alloy to 3.2% Co alloy indicated that similar segregation, but varying in amount, was present in all alloys. Figures 9 and 10 are optical micrographs showing several fields of view. Notice obvious Co phases are present in nearly every particle. However, a few particles still contain large amounts of Co_2Al_9 dispersed as a massive phase, which indicates Co segregation within the melt. A bottom pouring mechanism is used on the AGT 500000 rig and is such that Co segregation to the bottom of the melt could allow the formation of high Co containing powder particles. Additionally, the temperature of the nozzle/transfer tube was maintained below the liquidus of the alloy. The temperature is within a range that could allow primary particles of Co_2Al_9 to precipitate within the liquid prior to atomization. This problem is being actively investigated at this time.

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Etch: Kellers

A
Longitudinal

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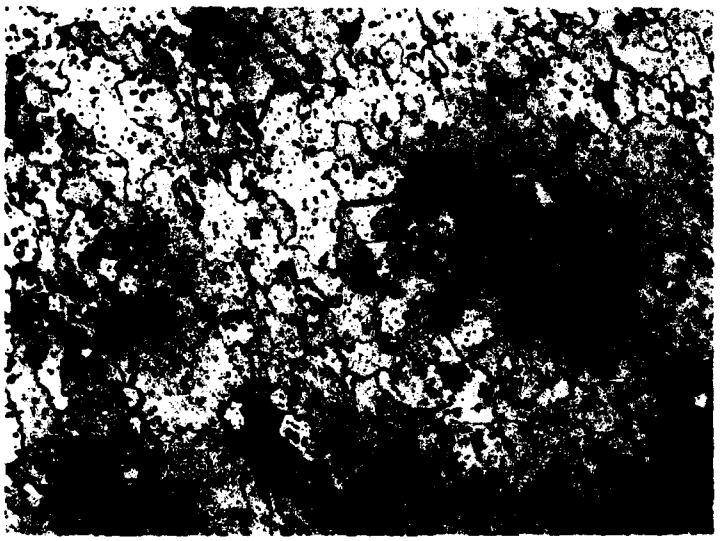

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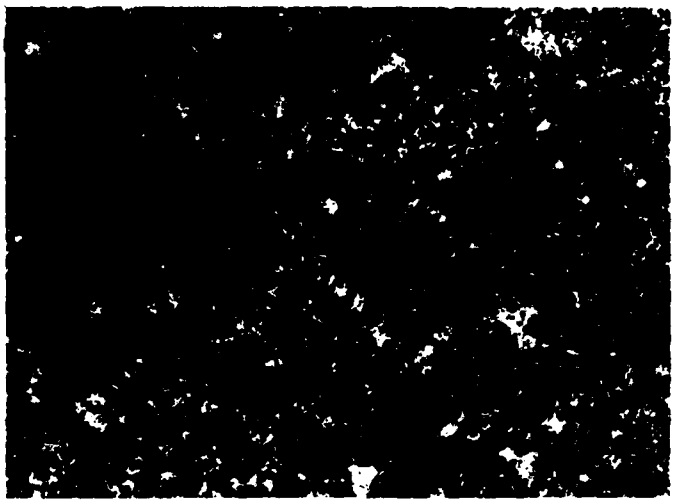
Etch: Kellers

B
Transverse

FAM 89119
FD 151991

Figure 1. Microstructure of Conventionally Processed Al 7075-T6



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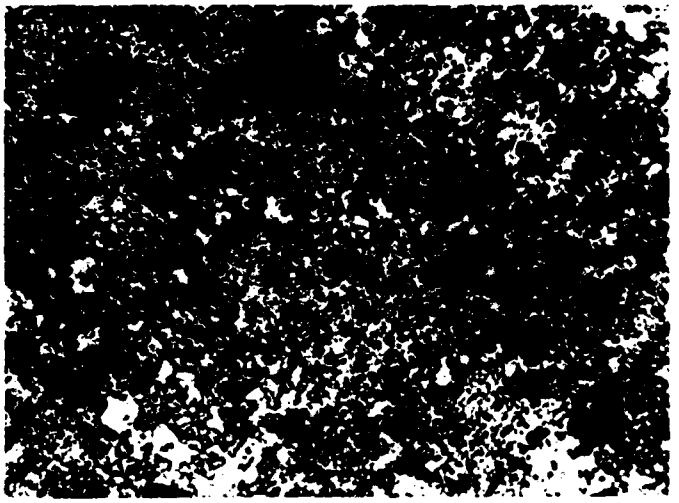
FAM 89493

A
Transverse



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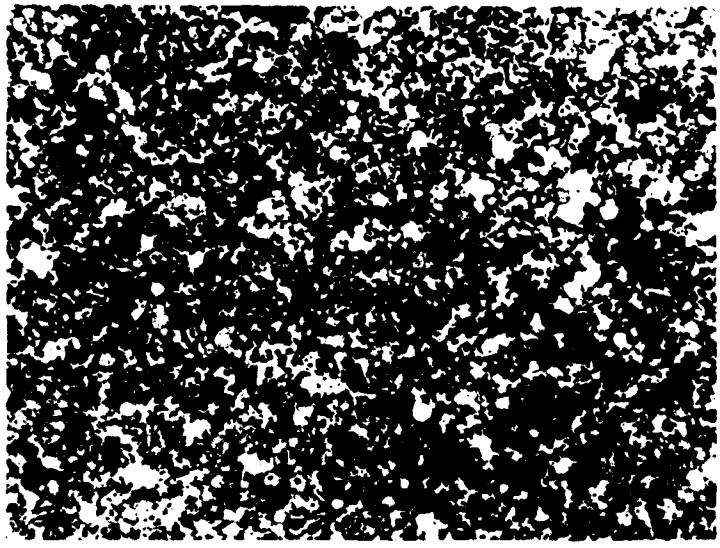
B
Transverse

Figure 2. Microstructure of XSR No. 40, Heat Treated



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A
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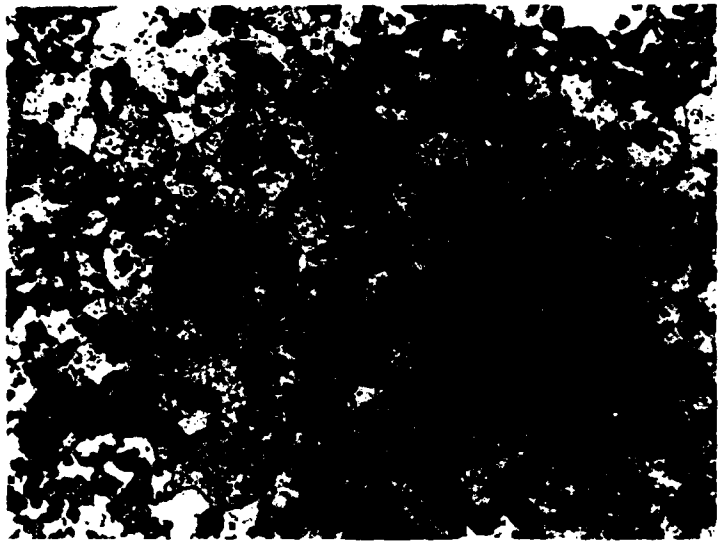
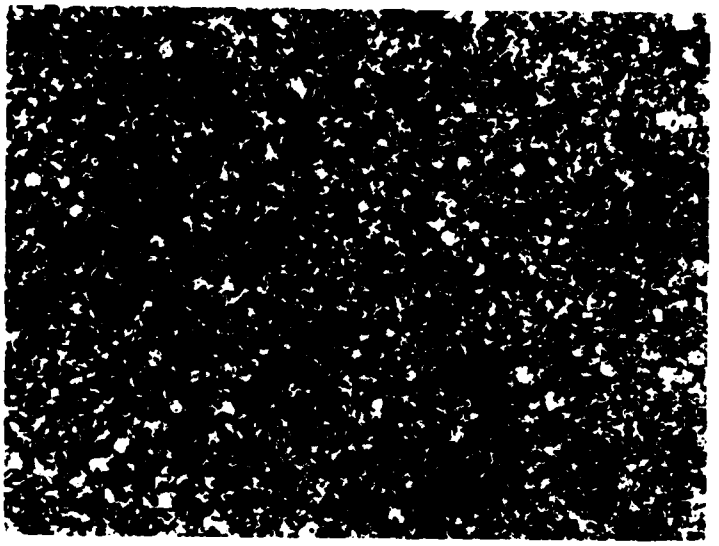


Mag: 400X
Etch: Kellers

B
Transverse

FAM 89498
FD 163049A

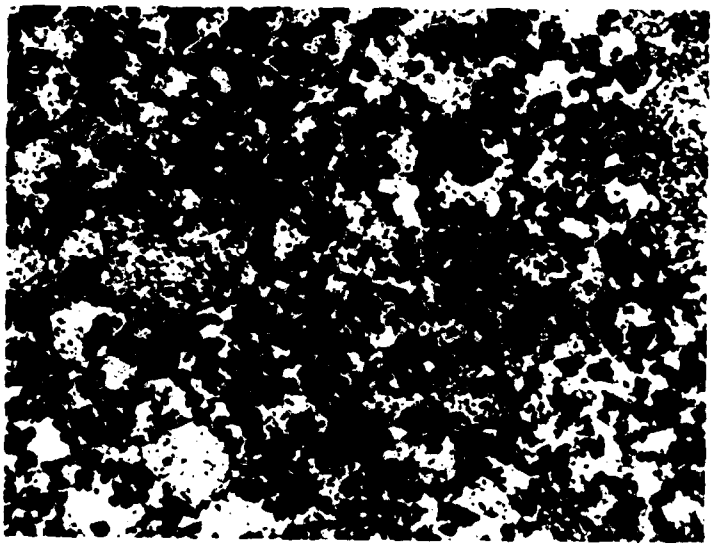
Figure 3. Microstructure of XSR No. 44, Heat Treated



FAM 89501

Mag: 100X
Etch: Kellers

A
Transverse

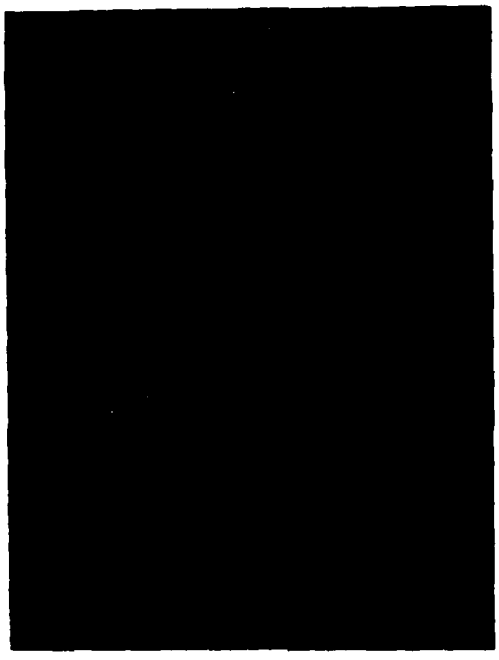


FAM 89502
FD 163050A

Mag: 400X
Etch: Kellers

B
Transverse

Figure 4. Microstructure of XSR No. 45, Heat Treated



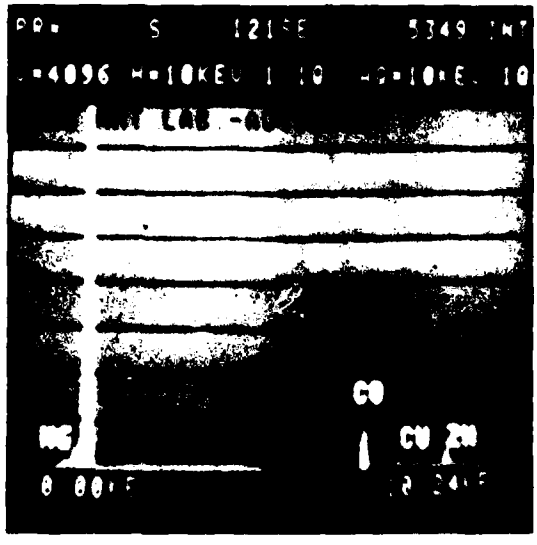
Mag: 400X A FAM 89220
Etch: Kellers Transverse

Mag: 400X B FAM 89239
Etch: Kellers Transverse FD 169037

Figure 5. Heat Treated Al Alloys Showing Massive Co_2Al_9 (1.6% Co); (A) XSR No. 36 Swaged, (B) XSR No. 37

Etch: Kellers FAM 89686

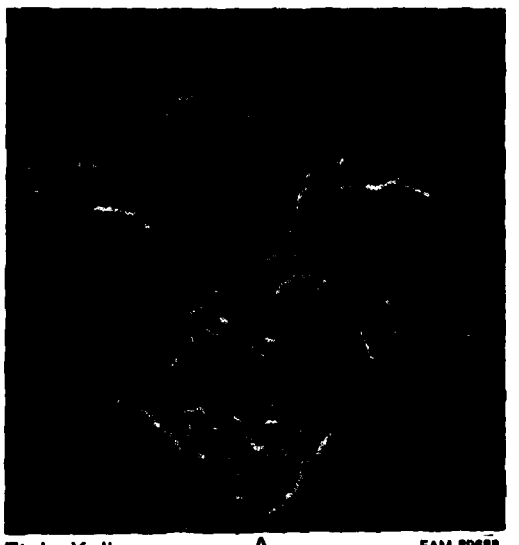
A



FAM 89687
FD 169038

B

Figure 6. (A) SEM Micrograph of Rapidly Solidified Power Particle (0.8% Co), (B) X-ray Scan Showing Alloying Elements



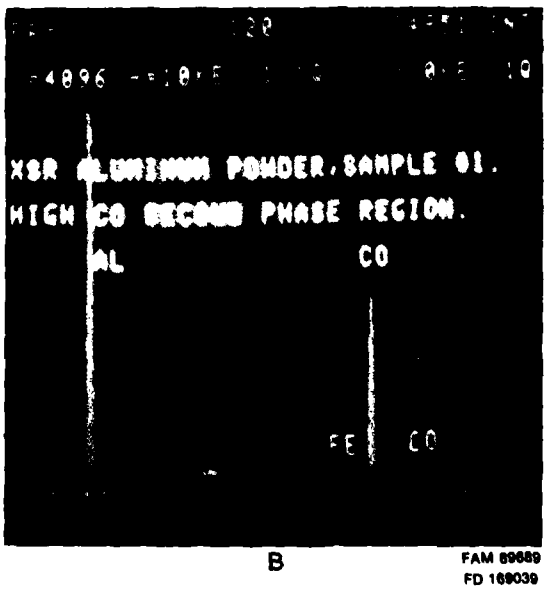


Figure 7. (A) SEM Micrograph of Co_2Al_9, (B) X-ray Scan of Second Phase Elements

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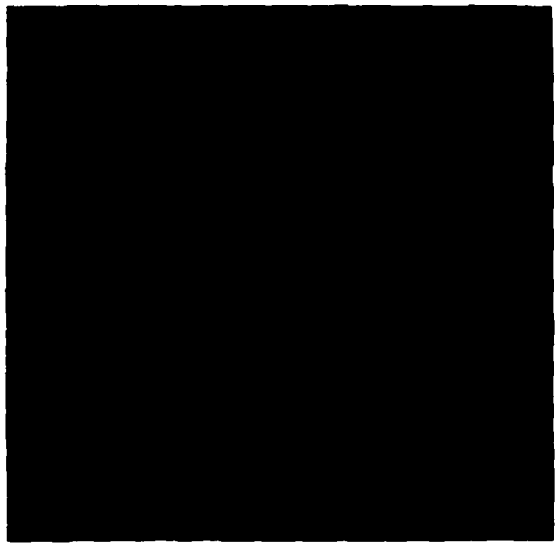

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FD 169040

Figure 8. (A) SEM Micrograph of Co Segregation in Rapidly Solidified Al Powder (0.8% Co) (B) Content Mapping of Co

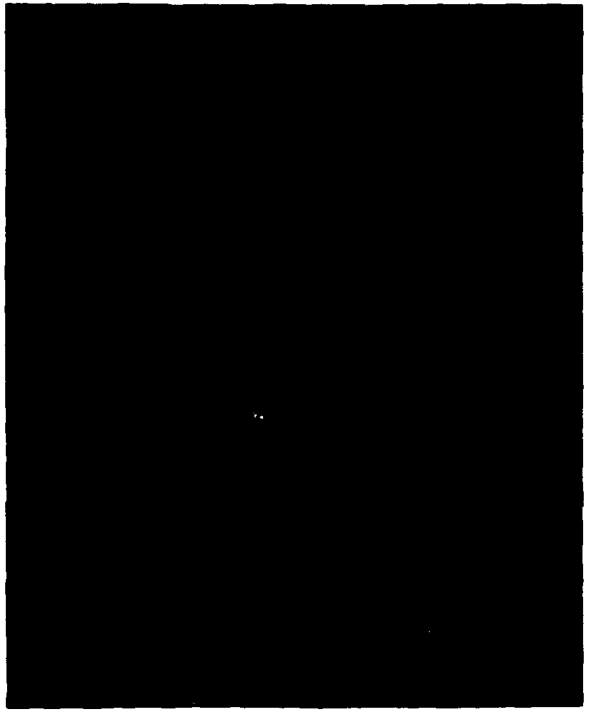


Figure 9. Rapidly Solidified Al Powder Alloy (3.2% Co)

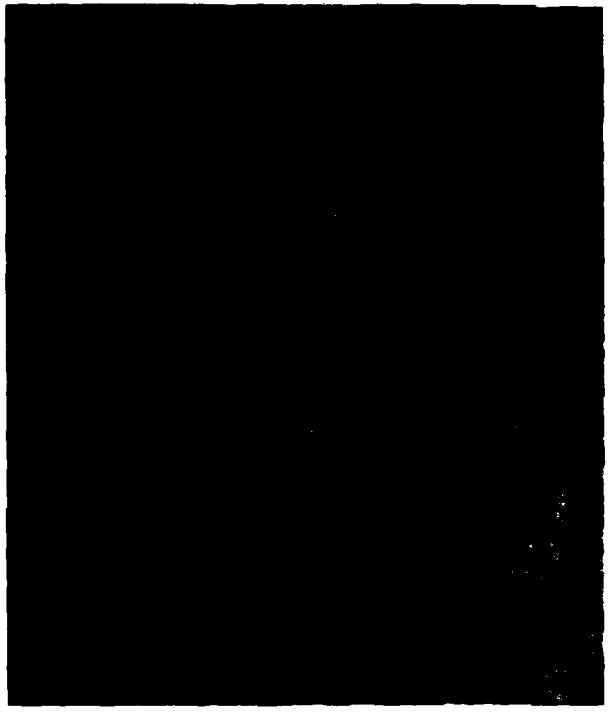


Figure 10. Rapidly Solidified Al Powder Alloy (3.2% Co)

Regardless of this segregation effect, hardness and tensile tests were carried out for these materials and for materials made previously. The data are listed in Table 6. Hardness values varied from somewhat above to somewhat below those values normally obtained for 7075-T6 material. In all probability, the variation is due to the segregation of the type noted above.

Tensile results, for the most part, were excellent. Every alloy tested, with one exception, showed strength levels exceeding the nominal value of 7075-T6 Al. The XSR No. 33 had a 38% improvement in yield strength over 7075-T6 while retaining good ductility of 8.0%. The increase in strength for these alloys seems to be mainly a grain size effect. Evidence to support this conclusion is shown in Figure 11. The two microstructures of XSR No. 30-1 shown come from the same length of extruded rod. Processing is the only difference between the the two rods. The normal processing is extrusion and a subsequent heat treatment, while the second bar was swaged at 750°F (399°C) prior to heat treatment. The result of this processing variation was a large difference in grain size and strength. The swaged rod was coarse-grained (ASTM 3) and had a yield strength of 69 ksi (475.4 MPa) while the fine-grained material (ASTM 9) had a yield strength of 93 ksi (633.9 MPa). All of the samples swaged exhibited these same differences when compared to extruded stock. Figure 12 shows a comparison of grain sizes for the various processing methods. There is an approximate 10% increase in yield strength for two of the three swaged XSR alloys when compared to 7075-T6, in addition to the strength increase due to grain size.

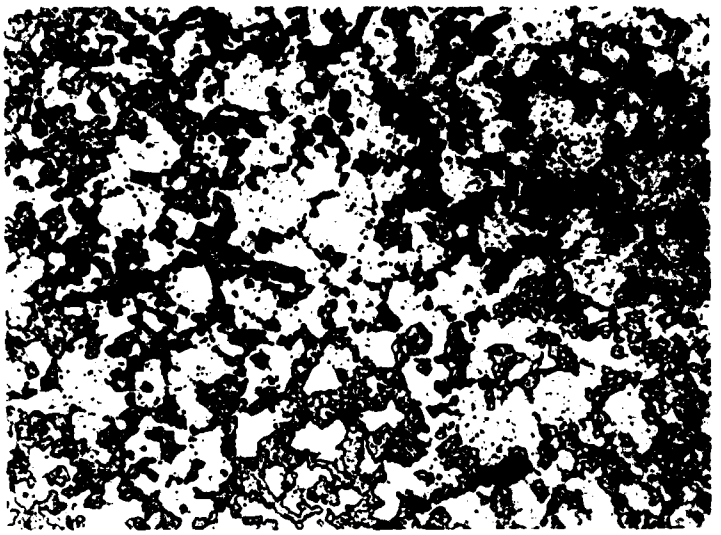
There appears to be a correlation between yield strength and extrusion temperature as shown in Figure 13. The strength level increases as the extrusion temperature decreases. This relationship is not necessarily linear as implied in the figure, and is only meant to show general trends. This applies not only to the average properties of the XSR alloys, but to individual alloys as well. Three alloy compositions which were extruded at more than one temperature, and in each case the material with the lowest extrusion temperature yielded the best properties.

IRON ALLOYS

As of yet, no evaluation of the most recent Fe alloys has been made. The emphasis was placed on conversion to powder and adaptation of XSR process to bearing steel applications. Evaluation of these alloys will be done the next quarter.

TABLE 6. ROOM TEMPERATURE HARDNESS AND TENSILE TESTS

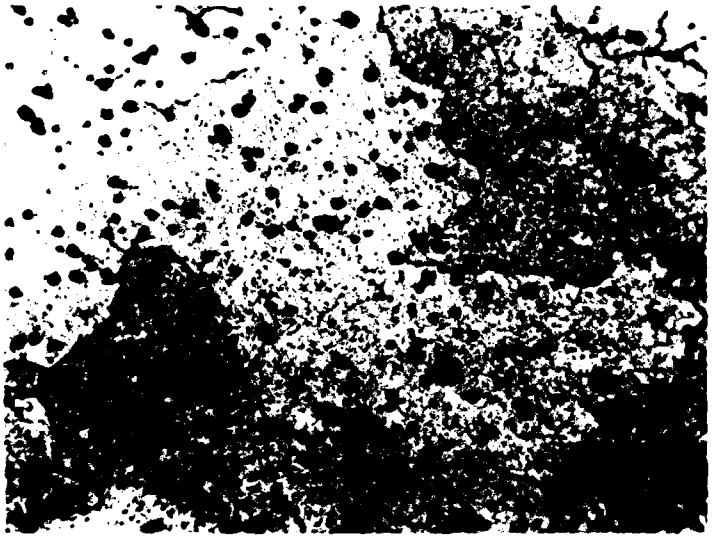
ID	*VM No.*	*0.2% Yield Strength*	*Tensile Strength*	*Elongation (%)*	*R_A (%)*	*Hardness R_B*	*Temperature °F (°C)*
7075-T6		68.4 (470.6)	82.4 (568.2)	15.1	29.0	90.0	
XSR-23	581	85.0 (586.1)	89.9 (619.8)	7.5		83.5	750 (399)
25	582	86.1 (593.6)	91.6 (631.6)	8.0		82.8	750 (399)
27	583	85.6 (590.2)	91.5 (629.9)	9.8	21.6	88.5	750 (399)
30-1	595	92.2 (635.7)	96.4 (664.7)	9.3		85.8	700 (371)
30-2	595	88.4 (609.5)	92.8 (639.8)	9.8		82.8	750 (399)
31	595	73.3 (505.4)	76.8 (529.5)	9.3		83.0	800 (427)
33	615	100.5 (693.0)	103.9 (716.6)	8.0	21.3	91.5	700 (371)
35	615	90.8 (626.0)	93.6 (645.4)	4.0		81.8	750 (399)
36	617	98.0 (675.7)	100.8 (695.0)	6.7	16.2	86.3	700 (371)
37	617	77.6 (535.1)	85.6 (590.2)	4.0	8.9	86.3	750 (399)
30-1S	595	69.2 (477.1)	78.5 (541.3)	14.7	28.8	90.3	Swaged 750 (399)
33-S	615	83.3 (574.4)	88.9 (612.9)	10.0	26.7	92.5	Swaged 750 (399)
36-S	617	80.7 (556.4)	87.4 (602.6)	9.7	22.6	90.2	Swaged 750 (399)
40	622					95.6	700 (371)
44	630					94.0	700 (371)
45	631					94.2	700 (371)



Mag: 400X
Etch: Kellers

A
Extruded
Transverse

FAM 89115



Mag: 400X
Etch: Kellers

B
Swaged
Transverse

FAM 89122
FD 158450A

Figure 11. Microstructure of XSR No. 30-1, Heat Treated

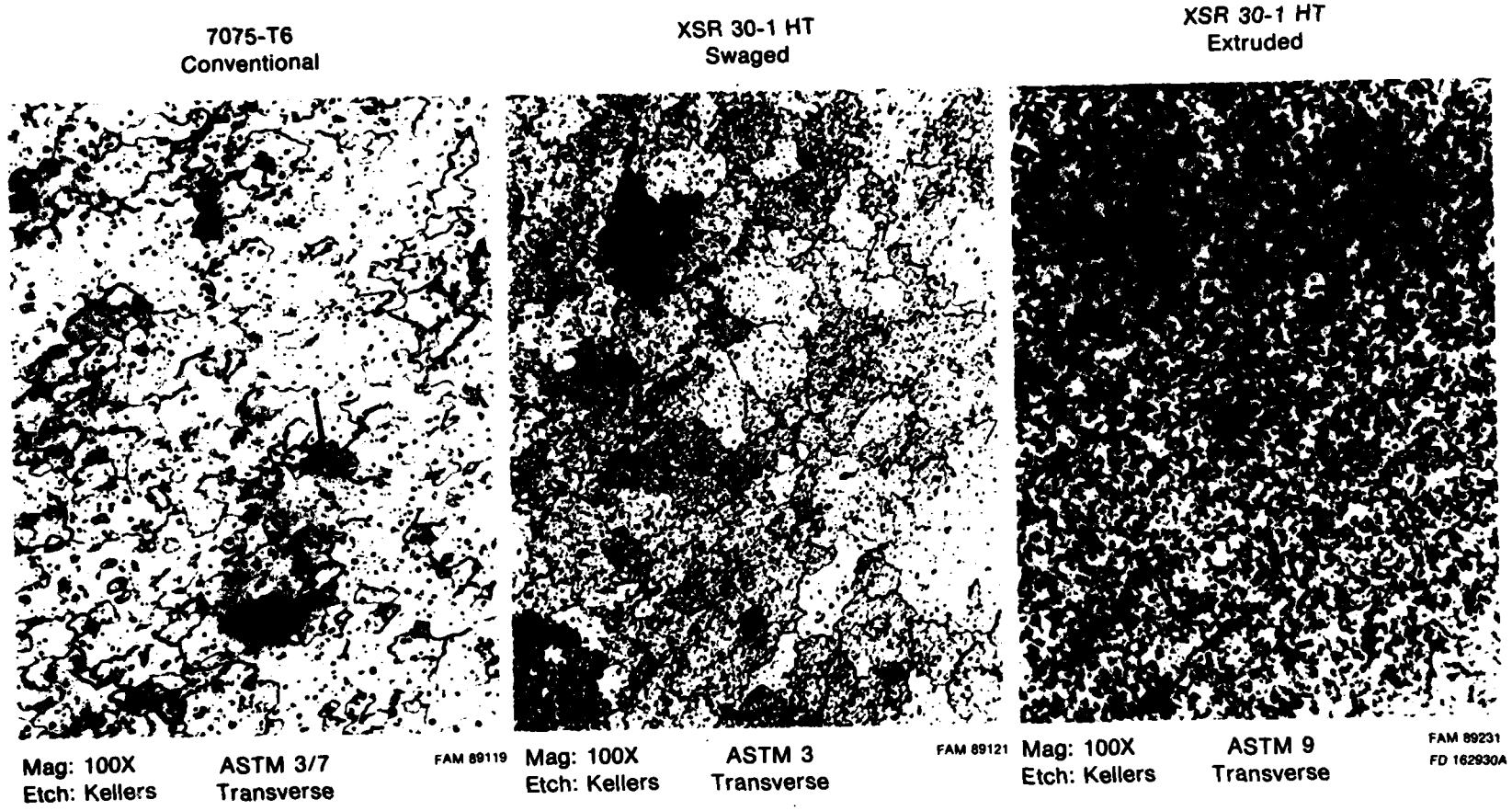


Figure 12. Effects of Processing of Grain Size

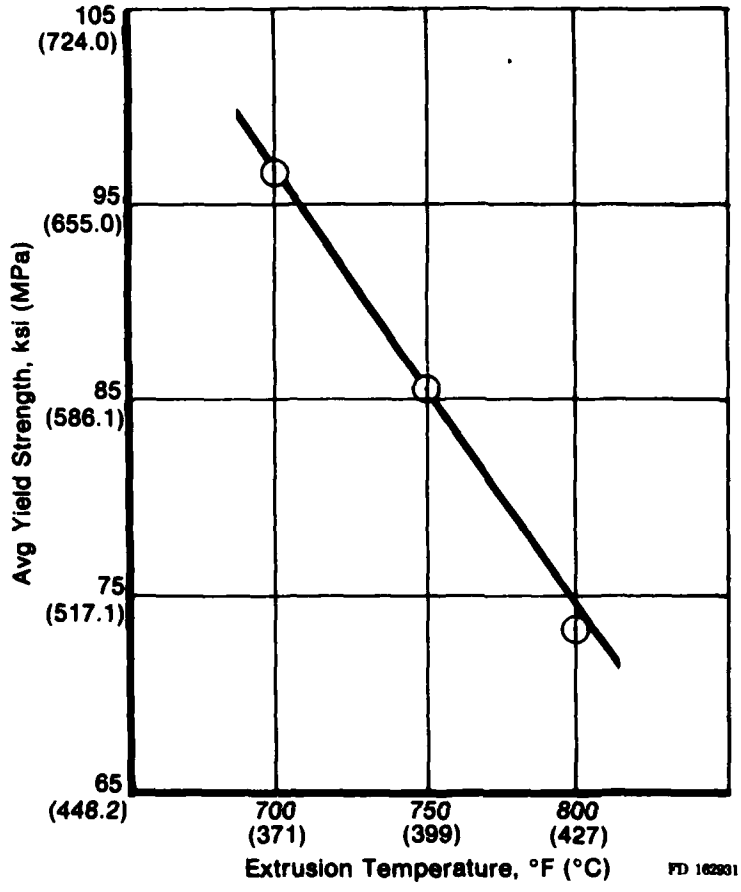


Figure 13. Effect of Extrusion Temperature on Yield Strength

SECTION V

ONGOING STUDY

In the next quarter additional mechanical testing will be performed on both alloy systems. Powder conversions will also continue. A new Al matrix will be developed, and stress corrosion testing will be initiated. Extrusion will also be done for further evaluation of alloys. Additionally, a major effort will be undertaken to obtain a more uniform and finer dispersion of Co_2Al_9. Higher pour temperatures and a higher nozzle temperature through furnace redesign will be accomplished and their effects evaluated.

SECTION VI

DIFFERENTIAL THERMAL ANALYSIS OF RAPIDLY SOLIDIFIED ALUMINUM ALLOYS

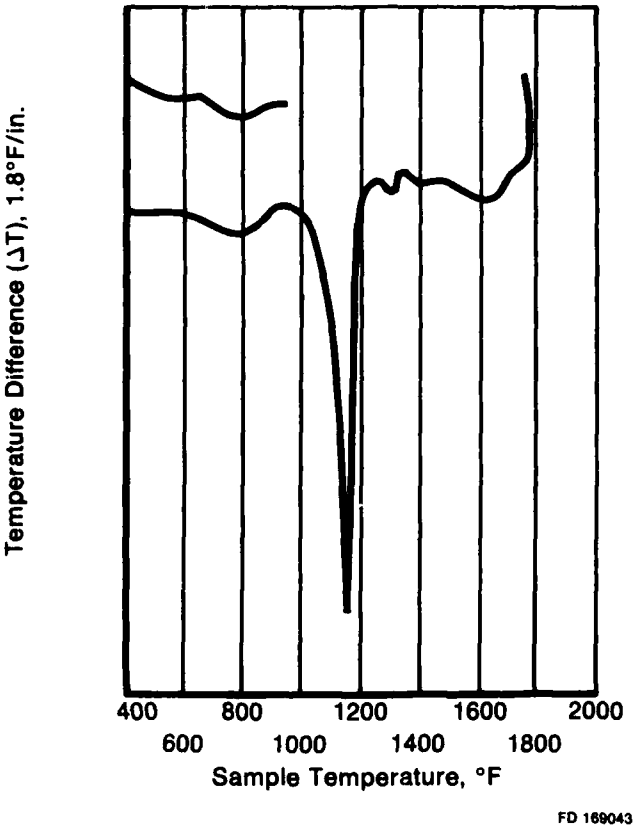


Figure 14. XSR No. 40, DTA Trace of Powder Sample. The Upper Curve is the Original Trace, the Lower Curve is a Reheat of the Same Sample

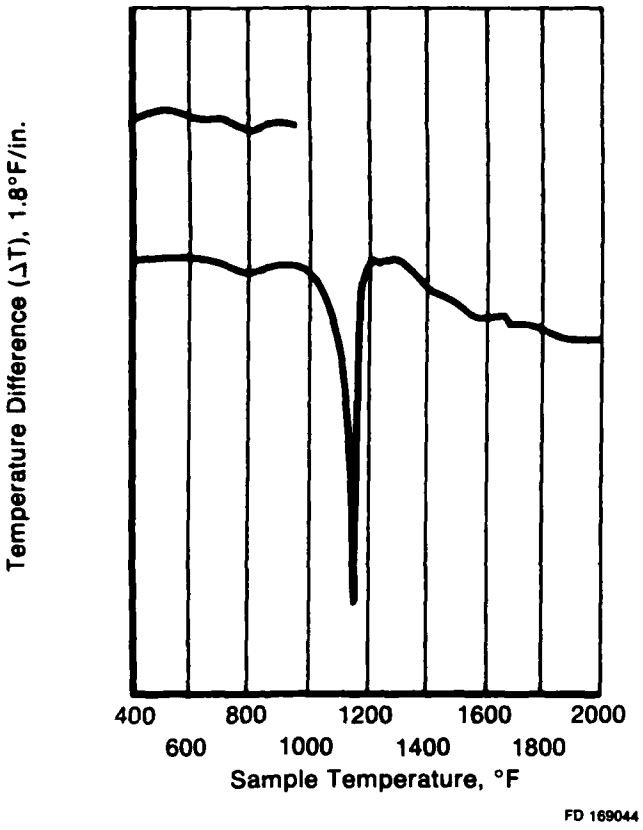


Figure 15. XSR No. 44, DTA Trace of Powder Sample. The Upper Curve is the Original Trace, the Lower Curve is a Reheat of the Same Sample

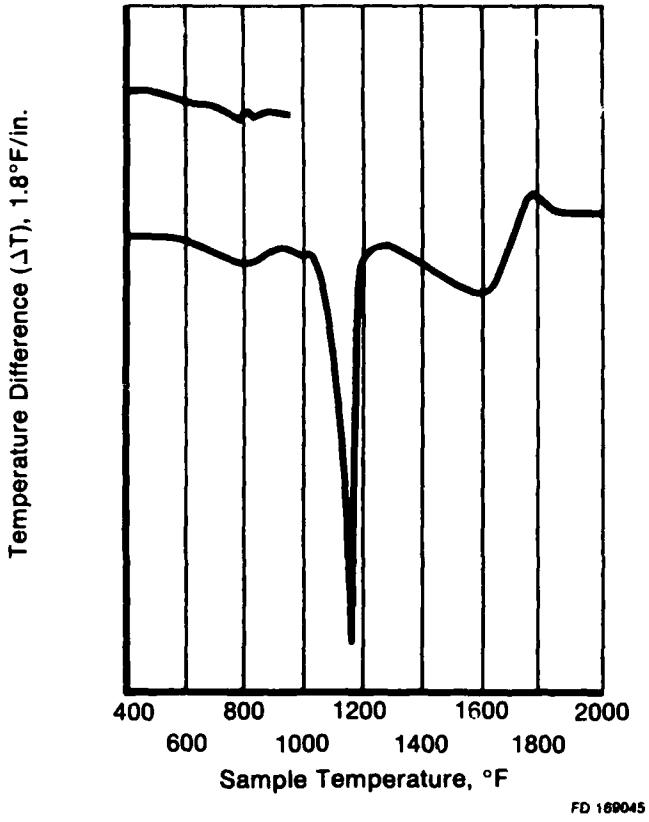


Figure 16. XSR No. 45, DTA Trace of Powder Sample. The Upper Curve is the Original Trace, the Lower Curve is a Reheat of the Same Sample

END

FILMED

1-85

DTIC